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TECHNICAL REPORT NO. 5

Corrosion of the Zinc Electrode in the Silver-Zinc-Alkali Cell

by

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#### ABSTRACT

A study has been made of the factors that affect or bring about the corrosion of the zinc electrode in a silver-zinc-alkali cell. Cells containing 30% potassium hydroxide as electrolyte were used and kept at room temperature. Special attention was centered on open circuit or stand conditions. This corrosion is affected primarily by oxygen and by dissolved silver oxides.

#### INTRODUCTION

Although the so-called silver-zinc-alkali cell has been produced for a relatively short time its applications are increasing and it is becoming a rather widely use system. It is now being marketed as a secondary cell having a fairly good life. It is especially remarkable that these advances and developments have been made without a thorough understanding of the mechanisms of the electrode reactions. Relatively little work of a more or less theoretical or fundamental nature has been reported on this system.

In a previous report (1) it was noted that the anodic dissolution of zinc in potassium hydroxide solutions proceeds in two steps. However, when this reaction takes place in a silver cell the mechanism is undoubtedly more complex since there is then the additional complicating factor of the presence of the silver oxides. It was the purpose of this study to investigate the effect of silver oxides and other factors on this anodic zinc process.

The effect of the silver oxides on the anodic dissolution of zinc in potassium hydroxide solutions is especially obvious when one studies the relationship of this process to Faraday's laws. Two solutions, 15% and 30% potassium hydroxide were used as the electrolyte in cells containing zinc anodes and nickel screen cathodes. Two silver coulometers were connected in series with these cells and a small current density was used for about two hours. Comparing

the amount of zinc oxidized (dissolved) with that prescribed by Faraday's laws, there were deviations of +4% and +0.1%. When a similar test was made on a cell containing a silver oxide cathode in place of the nickel screen, and 30% potassium hydroxide as the electrolyte, the deviation was +93%, i.e., the loss of zinc was about twice as great as that expected from Faraday's laws. A good deal of this loss was due to the fact that during passage of current a heavy coating of spongy material formed on the anode (zinc) and fell off when the electrode was removed from the solution. This latter fact, aside from the deviation from Faraday's laws, prompted a further investigation of this process or phenomenon. A similar phenomenon was produced under open circuit conditions and X-ray analysis showed that the spongy metallic product was the same in each case. Therefore an attempt was made to determine the origin and nature of this deposit. This was done by using only open circuit conditions.

#### EXPERIMENTAL

Samples of zinc sheet, better than 99.9% pure, furnished by the New Jersey Zinc Sales Co., were cut into strips about 1 cm. wide. These were degreased and then treated briefly with diluted hydrochloric acid. In some cases such a zinc electrode, together with another electrode made by pasting silver oxide on a silver screen, was placed in a large test tube containing about 50 ml. of 30% potassium hydroxide solution. The electrodes were electrically insulated from each other. After being in the cell for twenty four hours, the zinc was withdrawn, briefly immersed in 6 N acetic acid, then in distilled water, in ethanol, and finally in acetone. Following this the electrode was dried by heating for a short while. Each run was carried out in a constant temperature bath held at  $25 \pm 0.1^\circ\text{C}$ . The loss of zinc was determined by the difference in weight. The surface area of the immersed zinc was about 10 sq. cm. In some runs the

silver oxide electrode was omitted, and in others a smaller test tube and a smaller volume of electrolyte were used. X-ray diffraction patterns were obtained by the use of copper radiation with a nickel foil filter.

#### RESULTS

It soon became apparent that even on open circuit several factors were involved in the corrosion of the zinc. When cells were assembled using silver oxide, the zinc sheet, and 30% potassium hydroxide solution, the zinc electrode always became covered with a dark black film. This film had a velvety appearance when viewed with a microscope. A black film is sometimes noted on the zinc electrodes after a discharge and then it is often due to a form of zinc oxide (1). In this case, however, the black film did not appear to be the zinc oxide. It did contain varying, but appreciable, amounts of silver or its oxides. This was noted by dissolving a portion of the electrode in nitric acid and then adding hydrochloric acid to precipitate the silver as the chloride. X-ray diffraction patterns obtained from this film contained the lines due to zinc, but there were other additional lines as well, particularly at about 2.25 and 2.15Å. These extra lines were found in all such samples. Furthermore, these same lines were found in the spongy material that peeled off the zinc electrode when it was removed from the solution after having received a twenty four hour anodic treatment. They were also produced by the dark colored film that forms on the zinc shortly after the discharge of a silver oxide-zinc-alkali cell is begun. These two lines are not due to silver or its oxides. The indications then, are that although silver or its oxides were found on the zinc electrode, they are not present in appreciable quantities as metallic silver or oxides of silver. This black film was formed only when the silver oxide was present in the cell, not when zinc alone was immersed in the potassium hydroxide solutions. Thus the silver oxide electrode is in some manner related to this film on the

zinc. However, the effects of other conditions were also studied.

Table I gives a summary of the results obtained. Column 2 indicates the conditions under which each run was made. In some runs no electrode of silver oxide was present, but rather some silver oxide powder was placed in the test tube with the electrolyte and the zinc was placed on glass beads in the bottom of the test tube. This is indicated in column 3. In other cases the zinc specimen was given a silver coating by dipping it in a silver nitrate solution before it was assembled in the cell. This is indicated in Column 5. Column 7 refers to the nature of the coating formed on the zinc specimen.

From these results several conclusions can be drawn as to the nature of the process taking place. First of all it appears that under the conditions prevailing in these experiments the amount of electrolyte had but little effect, see runs 17 and 21.

A significant factor is oxygen, either dissolved in the electrolyte, or present in the atmosphere above the electrolyte. There is no doubt that such oxygen affects the corrosion of zinc. In these experiments white deposits of zinc oxide were sometimes formed at the electrolyte surface. This oxide is soluble in potassium hydroxide solutions and hence such oxide formation offers no protection against corrosion. The effect of oxygen is noted by comparing runs 1 with 3 and 2 with 4. In each pair all the conditions were alike except for the bubbling of air through the electrolyte. However, even when air was not bubbled through the solution the effect of oxygen could be noted. Comparison of runs 4 with 5, 10 with 11, and 13 with 14, shows the effect of dissolved oxygen. In runs 5, 11, and 13, the electrolyte was not fresh. Consequently, dissolved oxygen had already been consumed. Comparing these with otherwise identical runs it is seen that in each case the corrosion of

TABLE I

FACTORS CONTRIBUTING TO THE CORROSION OF ZINC IN 30% KOH AT 25°C.

No.	Conditions*	Silver present as			wt. loss of zinc in mg.	color of film
		silver oxide powder in electrolyte	silver oxide electrode	metallic coating on zinc		
1	a, g		x		337	spongy
2	a, g, g		x		130	black
3	g		x		52	black
4	e, g		x		55	black
5	c, f, g		x		26	black
6	b, g, g		x		136	black
7	b, c, g		x		145	black
8	e, g		x		138	black
9	h	x			18	light black
10	h	x			31	light black
11	f, h	x			3	light black
12	d, h			x	78	clear
13	d, f, h			x	29	clear
14	d, h			x	65	clear
15	a, g				46	clear
16	h				39	clear
17	h				43	clear
18	b, g				8	clear
19	e, h				31	clear
20	d, h				6	clear
21	g				43	clear

\* the various conditions are:

- a- air bubbled through electrolyte throughout experiment.
- b- nitrogen bubbled through electrolyte throughout experiment.
- c- electrolyte saturated with zinc oxide.
- d- zinc completely submerged, i.e., no "water line" effect.
- e- electrolyte contains 0.2% hydrogen peroxide.
- f- electrolyte was not fresh but had just been used for another run.
- g- 50 ml. of electrolyte used.
- h- 6 ml. of electrolyte used.



zinc was considerably less with the oxygen-deficient electrolyte. This was true whether silver oxide was present or not.

Still another evidence for this is found by comparing runs 17 and 20. In 20 the zinc was completely submerged whereas in 17 part of the zinc was out of the electrolyte. The zinc was attacked to a greater extent in the latter case where atmospheric oxygen as well as dissolved oxygen was available for reaction. Furthermore, in run 18 nitrogen was bubbled through the electrolyte thus removing the oxygen. Here again the weight loss of zinc was less than in run 21.

Thus the oxygen does attack the zinc. The mechanism of this reaction probably involves local cells containing zinc and oxygen electrodes. In effect, the oxygen transforms the zinc to zinc oxide or hydroxide which then dissolves in the potassium hydroxide solution. If this is the course of the process, then if the electrolyte were saturated with zinc oxide the oxidation product of the zinc would not dissolve as readily and this product might then offer some protection to the remaining zinc. In run 2 and electrolyte saturated with zinc oxide was used and the attack on zinc was definitely less than in run 1. In both these runs air was bubbled through the electrolyte, thus heightening the effect of oxygen. Where this was not done the effect of zinc oxide-saturated potassium hydroxide was negligible, e.g., runs 3 and 4, runs 6 and 7.

However, under certain conditions, passing air through the electrolyte made very little difference. In run 15 the loss in weight of zinc was about the same as that in run 21. This may indicate that the oxygen attack is primarily by the dissolved oxygen. This difference may have been more pronounced if the run had continued longer.

Oxygen attacks and corrodes the zinc electrode but this attack by itself

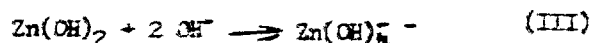
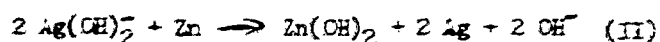
does not produce the black film referred to earlier. Silver oxide is necessary for this and it produces the black film even in the absence of oxygen, run 6. Thus the actions of oxygen and of silver oxide are different, but they often occur simultaneously.

The effect of silver oxide electrodes on the corrosion of zinc is readily seen by comparing runs 1 with 15; 3 with 21; and 6 with 18. In each case the corrosion of zinc was greater in the presence of the silver oxide electrodes. The difference was not the same in each of these three cases. The reasons for this are: (a) The weight loss is not accurate in each case since, especially in run 1, a large amount of spongy metallic material was formed which fell off when the electrode was removed; (b) In some cases the electrolyte was agitated with air or with nitrogen. When no agitation was used, runs 3 and 21, the difference was significant but not large. When the electrolyte was agitated by bubbling nitrogen through it, the corrosion loss of zinc due to the presence of silver oxide electrodes was greater, runs 6 and 18. In each case the zinc received a velvety black coating or a spongy metallic deposit.

An explanation for this phenomenon involves the ready dissolution of silver oxide in potassium hydroxide solutions. This is shown on Figure 1. Small amounts of silver oxide were placed in stoppered flasks containing 25% potassium hydroxide solution. These mixtures were kept at room temperature, shaken occasionally, and from time to time samples were withdrawn, filtered through glass wool, and analyzed for silver by titrating potentiometrically with a potassium iodide solution. The results show that the silver oxide dissolves rapidly in potassium hydroxide solutions. When zinc was added to these solutions the dissolved silver was removed rather rapidly.

Quite likely then, the silver oxide from the electrode dissolves in the potassium hydroxide electrolyte. When this dissolved silver reaches the zinc

electrode it reacts with the zinc and precipitates there, the reactions probably being



This mechanism accounts for the presence of silver found in the black film or metallic deposit on the zinc specimens. It also accounts for the effect of agitation, e.g., comparing run 3 with 6 where no oxygen was present but the solution was agitated with nitrogen. This agitation hastens the diffusion of silver oxide to the zinc specimen and thus reaction (II) takes place more rapidly. Comparison of run 4 with 7 supports this hypothesis.

Further support for this mechanism is found in the fact that the appearance of the dark film on the zinc when the silver-zinc cell is discharged is more rapid as the temperature increases and as the concentration of the potassium hydroxide increases. Increasing the temperature would increase the solubility of silver oxide and also the rate of diffusion of dissolved ions. The solubility of silver oxide also increases with increasing hydroxyl ion concentration (2).

When a little silver oxide was added to the electrolyte in the absence of silver oxide electrodes, the effect was slight, runs 9 and 10. This was undoubtedly due to the fact that the silver oxide rested on the bottom of the test tube with the zinc specimen suspended above it. In this case the dissolved silver oxide would have had to diffuse upward. That some of it did was shown by the fact that the zinc did become covered with a light black film. This was heaviest at the bottom of the electrode.

The dissolved silver oxide is largely in the form of a negative ion. Therefore, it is obvious that reaction (II) will be hastened during the discharge of the cell, since then the zinc functions as the anode. The silver ions will

then migrate more rapidly to the zinc. For this reason too the same kind of film is formed on the zinc during discharge of the cell as during stand.

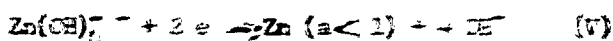
There still remains the question of the origin of the spongy metallic deposit on the zinc, especially in run 1. As has been noted, this deposit contained silver but no silver lines were found in the X-ray pattern. Instead there were zinc lines plus others. A possible mechanism for this phenomenon is given below.

When reaction (II) takes place it may do so only at certain sites in the zinc lattice. The reduced silver then replaces the oxidized zinc atom. This gives the ordinary <sup>silver</sup> lattice with silver atoms substituted for zinc atoms at certain points. The result is a solid solution of silver in zinc. Many studies have been made of the silver-zinc system. Of particular help is the work of Westgren and Phragmen (3), Owen and Pickup (4), and Owen and Edwards (5), where the zinc-rich phases are discussed. Westgren and Phragmen give much information on the X-ray patterns of this system. The designation of the various phases differs so such phases will be designated here by composition.

On Figure 2 are given the X-ray lines for zinc, some silver-zinc alloys or solid solutions, and for a representative film obtained in our work. Comparing these patterns, it appears that the black film on the zinc specimens consists of zinc plus some silver-zinc solid solution having a composition varying from 50 to 80% zinc. The lines in Figure III-b vary somewhat with the amount of zinc present (3). The region 51 to 60% zinc consists of a mixture of the two phases represented in Figures III-b and III-c (4). These X-ray data lend support, then, to the hypothesis that when the dissolved silver oxide reacts with the zinc, a solid solution of silver in zinc is formed on the surface of this zinc.

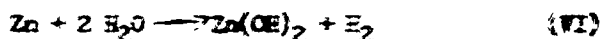
Furthermore, with this hypothesis one also has an explanation for the spongy metallic deposit formed on the zinc specimen in a few of the runs. It has been

Noted, analysis showed that this deposit contained zinc and silver. The formation of the silver-zinc solution lowers the activity of the zinc on the electrode at that site. The potential of the dissolution of zinc, equation (IV), would then be great enough to cause reaction (V) to take place at another part of the same

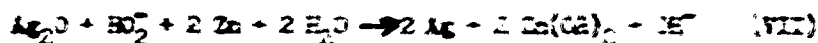


electrode. Another explanation is that suggested by Suranwaris and Pang (7) who studied a similar phenomenon in acid solutions. They suggested that the solid solution is less anodic than zinc and hence the solid solution can be deposited by the EMF of zinc itself.

There is also other evidence that metallic silver as such is not involved in this process. When zinc was covered with silver before being assembled in the cell, runs 12, 13, and 14, there was an increase in corrosion (compare with run 20) but there was no visible film on the zinc. Furthermore, when a silver electrode and a zinc specimen were electrically connected in a 30% potassium hydroxide solution the zinc dissolved and hydrogen was evolved, vigorously at first, on the silver but after twenty four hours no zinc appeared on the silver electrode. The reaction probably was



Two runs were also made to test the effect of hydrogen peroxide on this corrosion of zinc. The presence of the peroxy ion increases the corrosion of zinc considerably when silver oxide is present, see runs 5 and 8. In the basis of thermodynamic considerations the could be expected since the free energy decrease for reaction (VII) is about 25 kcal. greater than for reaction (VIII).



However, the mechanism is likely not as simple as indicated.

In the absence of silver oxide, the effect of hydrogen peroxide is less, see runs 17 and 19. In fact, the presence of the peroxy ion seems to reduce the corrosive loss of zinc. The effect, however, is small.

#### SUMMARY

In a silver-zinc-alkali cell the zinc electrode is attacked and oxidized by oxygen. When no diaphragm is present to reduce or eliminate diffusion, dissolved silver oxide migrates to the zinc electrode where it is reduced and forms a solid solution with the zinc, or else is deposited as such. This enables zinc or silver-zinc solid solution to precipitate from the electrolyte in a spongy form on the zinc electrode during open circuit conditions.

#### ACKNOWLEDGEMENT

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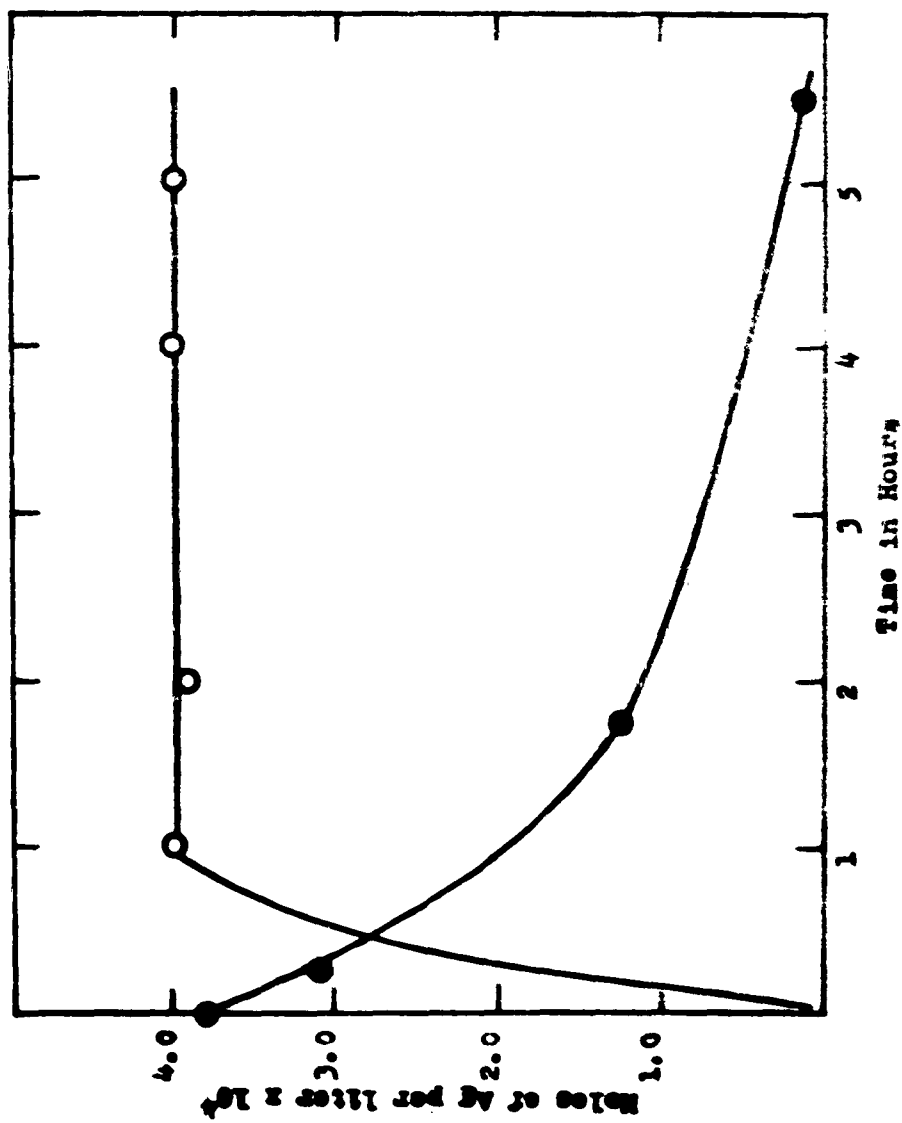


Figure 1



Figure 1. Dissolution and precipitation of silver oxide from 25% KOH solution. Open circles, dissolution of silver oxide; closed circles, precipitation of dissolved silver oxide by metallic zinc.

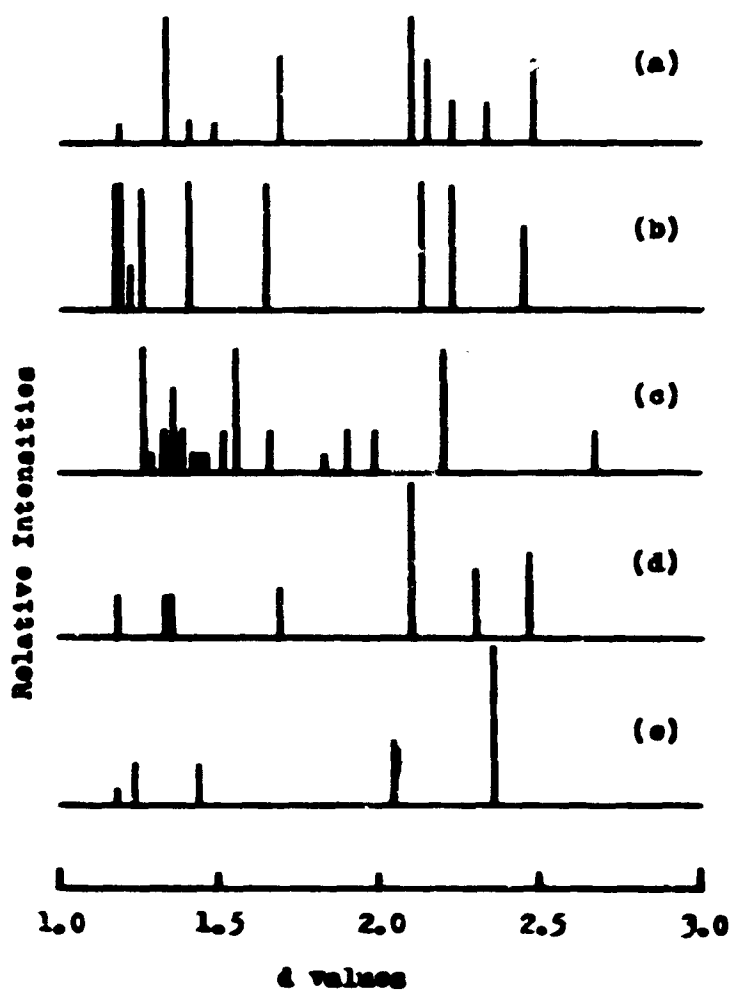


Figure 2

Figure 2. X-ray patterns: a) representative dark film obtained in this work; b) Ag-Zn solid solution ranging from 60 to 70% Zn, ref. (3); c) Ag-Zn, solid solution ranging from 48 to 51% Zn, ref. (3); d) Zinc, ref. (4); e) Silver, ref. (6).

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